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(54) Title: PROCESS AND DEVICE FOR SEPARATION OF HIGH-MOLECULAR COMPONENTS FROM THEIR SOLUTIONS

(61) Addition to: --

(62) Excerpt from: --

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(56) The following publications were taken into consideration for evaluating patentability:

DT-AS [examined] 1,120,138

FR Patent 1,242,020

PATENT CLAIMS

1. Process for the separation of high-molecular compounds from their solutions by means of water vapor, by introducing a flow of high-molecular compound in solution into a spraying device, where it will join with a flow of water vapor and then the high-molecular particles that form are separated from the surrounding medium, hereby characterized in that surface-active substances and, as the case may be, fillers, are added to the solution of the high-molecular compound and that the combined flows of water vapor and solution of high-molecular compound are fed into a cyclone.

2. Device for conducting the process according to claim 1, further characterized in that it is comprised of a screening device, a cyclone and a desorption vessel.

The invention concerns a process as well as a device for separating high-molecular compounds, particularly elastomers, which have been obtained by polymerization of olefins and diolefins, e.g., of ethylene, propylene, butadiene, isoprene, in aromatic or aliphatic hydrocarbons. The solutions obtained hereby contain, in addition to the elastomer, residues of polymerization catalysts, such as, e.g. vanadium, cobalt, and titanium salts, which are appropriately removed from the polymerization product during the separation of the elastomer.

Processes are known, in which the elastomers are separated by means of methyl alcohol, isopropyl alcohol or acetone, whereby the catalyst residues must

frequently be washed out. The disadvantages of this methodology lie in the drying of the products and the regeneration of the solvent.

Methods are also known, in which the elastomers are separated from the solution by means of water vapor. This methodology is based on a mechanical renewal of the surface of the elastomers and is conducted, for example, in a mixing device suitable for this.

Further, a method is known from the French Patent 1,242,020, in which rubber-type high-molecular substances are isolated from their solutions by introducing the solution into hot water in the presence of finely divided water-insoluble solid compounds and surface-active organic compounds. In this way, the solvent is distilled off, the polymer is separated in powder form and then purified by filtering and washing out.

Finally, a device is known from German Auslegeschrift [examined] 1,120,138, in which an elastomer solution is sprinkled into a chamber, whereby the polymer solution is combined with a water-vapor flow at right angles to the ejection direction. The elastomer is decomposed into small particles by the hot steam jet and coalesces with the evaporation of the solvent. Then, the elastomer crumbs are introduced together with additional water into a device, in which the crumbs are separated from the water by means of centrifuging or separating in the known way.

The task of the present invention is the effective separation of high-molecular compounds from their solutions.

This task is resolved according to the invention by a process, in which the high-molecular compounds are separated from their solutions by means of water vapor, by introducing a flow of high-molecular compound in solution into a spraying device, where it is combined with a flow of water vapor, and then the high-molecular particles that form are separated from the surrounding medium, characterized by the fact that surface-active substances and, as the case may be, fillers, are added to the solution of high-molecular compound and the combined flows of water vapor and solution of high-molecular compound are fed into a cyclone.

According to the invention, the high-molecular solution is mixed, for example, with an alcoholic solution of a nonionogenic emulsifier in a quantity of 1 part of emulsifier per 1000 to 5000 parts of the high-molecular compound.

In addition to the surface-active substances, fillers soot as rust or oil in a quantity of 100 parts of filler per 100 parts of dissolved elastomers are used in the case of elastomers.

According to the invention, together with the emulsifier or with the emulsifier and soot, the solution of high-molecular compound is introduced axially into the center of a spraying device, where it is combined with the tangentially directed flow of water vapor, followed by spraying, coalescing, and evaporation of the solvent. Then, the combined flows are fed into a cyclone or another device of this type, which has a sprinkling plant, and the solvent vapors are separated from the suspension of high-molecular substance in water. The presence of surfactants prevents a clumping of the coalescing material and further has the

consequence that the residual catalyst is better washed out. Simultaneously, the deposition of the elastomer onto the walls of the nozzle is prevented, and as a consequence of the increase in wettability, a better separation of the polymer molecules from the water-vapor flow is produced in the separation device. Preferably, a nonionogenic emulsifier, e.g., an addition product of ethylene oxide and cetyl oil alcohol is used.

The surface-active agent makes superfluous the quenching of the vapors by means of cold water and facilitates the basic washing out of the residual catalyst soluble in water. A pure polymer product is obtained hereby, which contains only traces of impurities on the order of a few up to several dozen parts per million. The aqueous suspension of the separated product, which contains approximately 1 to 2% solvent, is introduced into a desorption tank, whereby an intermixing is produced by blowing in a flow of water vapor, and the remainder of the solvent is removed. Appropriately, the discharged vapor from the mixer is fed together with the recycled water into the cylcone.

In order to conduct the process of the invention, a device is proposed, which is comprised of spraying device, cyclone and desorption vessel, and the separation of the elastomer, separation of the solvent and washing out of the residual catalyst is made possible in a simple way.

This device will be explained in more detail in the following on the basis of the drawing and a preferred example of embodiment.

Fig. 1 shows a spraying device 6, a cyclone 1, and a desorption tank 2.

The spraying device is comprised of a vapor discharge connection 7, a water sprinkling connection 8, and a discharge tube 9.

The desorption tank has a connection 10, through which the additional substances are introduced, a vapor introduction connection 11, a connection 12 for introducing circulation water, a connection 13 for discharging the coalesced mixture, an outflow connection 14, and a connection 15 for discharging the vapors.

Appropriately, a premixer finds use, in which the deactivation of the catalyst, for example, with alcohol or water is conducted, along with and stirring of the elastomer solution with appropriate quantities of surfactant and fillers, such as soot, naphthene oil, calcium stearate, [and] zinc stearate.

The separated elastomer leaves the device in the form of an aqueous dispersion and can be separated in conventional separation devices, such as, e.g. in vacuum filters, centrifuges, oscillating resonance screens, etc.

After discharge from the device, the solvent vapors are guided to a cooler and the solvent itself can be fed back into the polymerization process without additional cleaning, after separation of the water.

The spraying device according to the invention, which is represented in section in Fig. 2, is comprised of nozzle housing 2, into which the solution of high-molecular substance is introduced through connection 1, throttle blocking unit 3, shaft 4, which is made from a tube, with which the elastomer solution is introduced into spraying nozzle 5, a connection 6, through which water vapor is introduced, a water-vapor distribution chamber 7, a diffusor 8, in which spraying,

evaporating of the solvent, and coalescing of the elastomer occur, a hand wheel 9 and a flanged connection 10.

This type of construction of the spraying nozzle assures the formation of a vacuum region at the output from the nozzle, which favorably influences the influx of the elastomer into the spraying region, the evaporation of the solvent, and the coalescence. The mixture, which is comprised of solvent vapors, water and coalesced material, after leaving the spraying device, is introduced tangentially to the water layer into the vessel that is the cyclone, in which is produced the separation of water, dispersion and solvent vapors.

Example 1

The solution of an ethylene-propylene-diene terpolymer, which was obtained by anionic polymerization in toluene and contains 7.2 weight percent of elastomer, is reacted with a quantity of an alcoholic solution of an addition product of ethylene oxine and cetyl oil alcohol, such that 1000 parts of elastomer are used for one part of emulsifier. The alcohol decomposes the catalyst residues present in solution.

The solution prepared in this way is coalesced in a device according to the invention with the use of steam at 12 atmospheres.

The product separated in the form of an aqueous dispersion is filtered and dried in vauum.

The ash content of the product, which is comprised primarily of Al_2O_3, amounts to 0.10%.

The extent of washing out of catalyst residues amounts to 93%. The consumption of vapor per 1 kg of polymer is 15 kg.

Example 2

To a solution of ethylene-propylene-diene terpolymer, which was obtained by anionic polyermization in heptane, which contains 8.6 weight percent of elastomer, the following are added: 50 weight parts of active soot, 30 weight parts of naphthene oil, and an alcoholic solution of the addition product of ethylene oxide and cetyl oil alcohol. Then coalescence is conducted in the device according to the invention, whereby steam is applied at a pressure of 8.0 atmospheres. The product separated in the form of an aqueous dispersion, is filtered off and dried in a vacuum drying device. The ash content of the product amounts to 0.14 %. The extent of washing out of the catalyst residues amounts to 92 %. The consumption of steam amounts to 12 kg per 1 kg of rubber.

One page of drawings attached hereto.

One sheet of drawings

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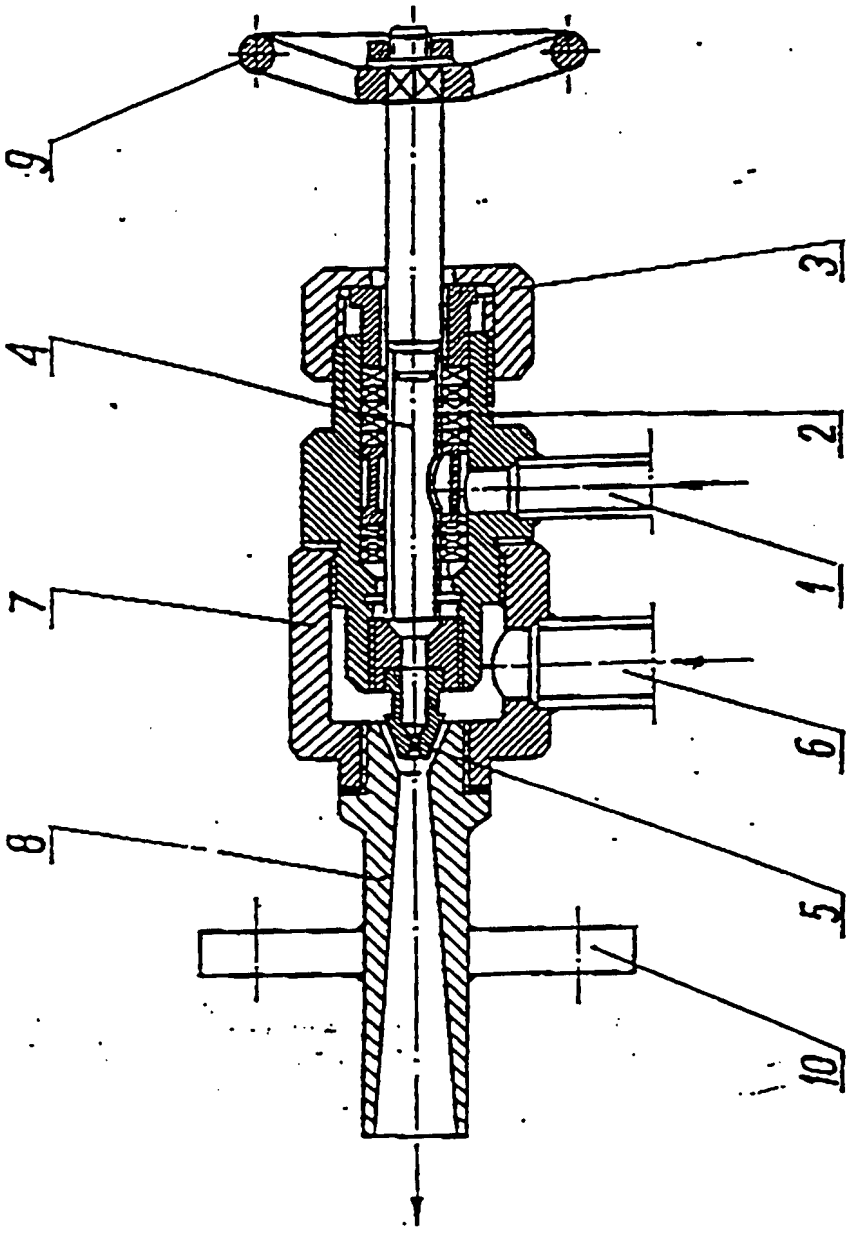

Fig. 2

One sheet of drawings

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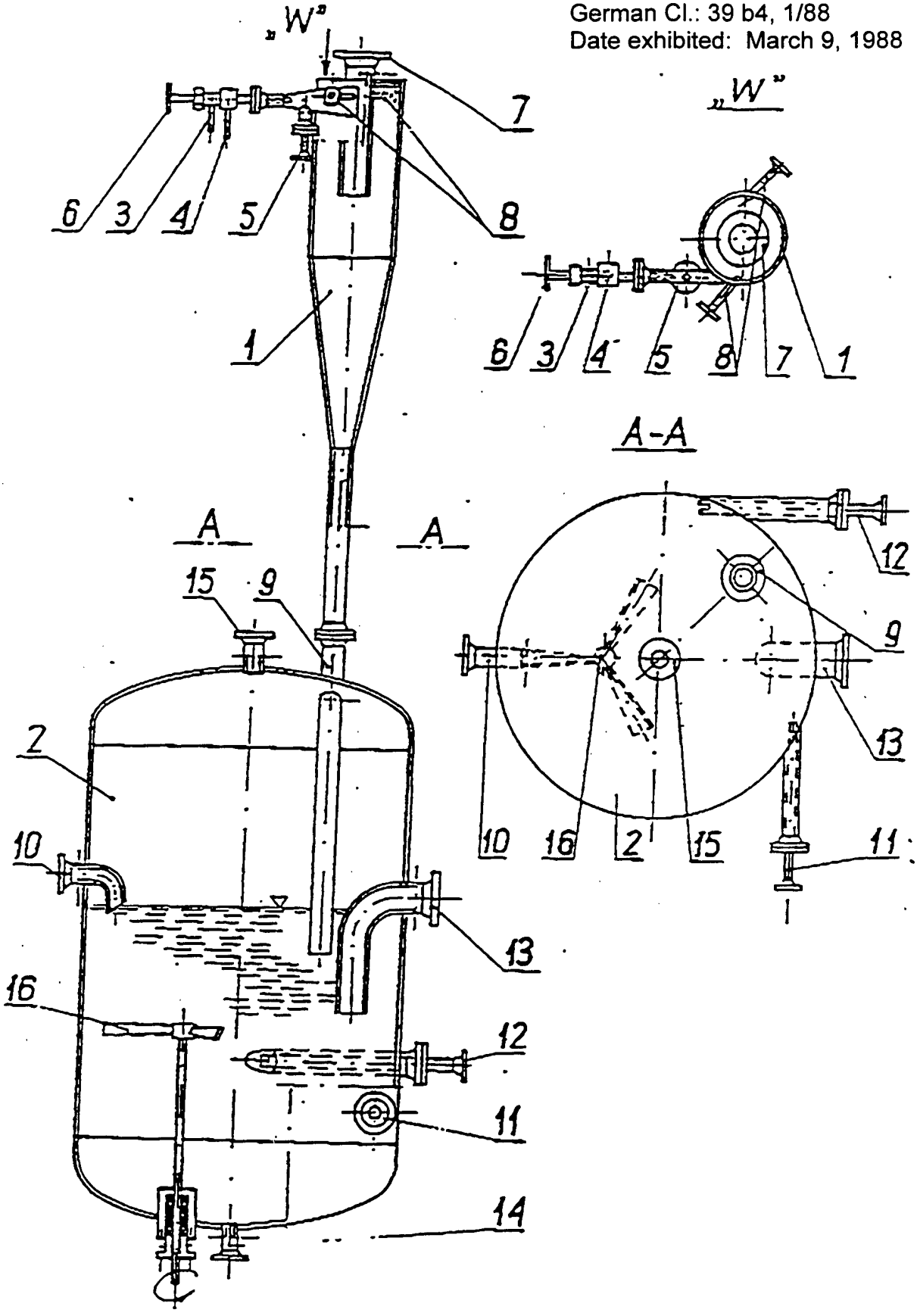

Fig. 1